

PATENT SPECIFICATION

337,123



Application Date: Sept. 6, 1929. No. 27,109/29.

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Complete Accepted: Oct. 30, 1930.

PROVISIONAL SPECIFICATION.

Improvements in the Production of Phosphorus Oxychloride.

We, JOHN STANLEY DUNN and FRANK BRIERS, both British Subjects, both of Norton Hall, The Green, Norton-on-Tees, County Durham, and IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W. 1, do hereby declare the nature of this invention to be as follows:—

10 It is known that when pure calcium orthophosphate is treated with phosgene at a temperature of 325—500°, phosphorus oxychloride distils over.

We have found that when most naturally occurring phosphates of calcium are treated with phosgene no substantial production of phosphorus oxychloride ensues. We have also found, however, that this absence of reaction is due to the formation of calcium chloride formed initially by the action of the phosgene upon the other calcium compounds present in the material, notably the carbonate. Based upon this observation, the invention consists in a method of obtaining from naturally occurring phosphates of calcium such as phosphate rock and the like, phosphorus oxychloride by treating the material to remove such other calcium compounds and thereupon submitting the purified calcium phosphate to the action of phosgene at a raised temperature.

For instance, phosphate rock containing 33 per cent. of phosphoric anhydride and 52 per cent. of lime, may be treated with phosgene at a temperature of about 300° until the carbonate present has been converted into chloride. During this treatment the termination of which may be ascertained by the disappearance

of carbon dioxide from the exit gases, no formation of phosphorus oxychloride is observed. The mass is then washed with water to remove calcium chloride and the washed and dried material is subjected to the renewed action of phosgene. An immediate production of phosphorus oxychloride occurs and up to 40 per cent. of the phosphate may be converted by the continued passage of phosgene. When the production of oxychloride slackens the mass may be leached with water to remove further calcium chloride formed by the reaction and the recovered phosphate used again.

The impurities in the crude phosphatic material may also be removed in other ways, for example by treatment with chlorine itself, following by leaching, or the crude material may be washed with a dilute acid such as hydrochloric acid or nitric acid.

Instead of using phosgene in the above process a mixture of carbon monoxide and chlorine may be used if the phosphatic material be mixed with activated carbon, which secures the formation of phosgene in situ. The carbon may be activated by preliminary treatment with chlorine at a high temperature, e.g. 1000° C. after which it is washed and dried before mixing with the raw material.

Dated this 6th day of September, 1929.

W. P. THOMPSON & Co.,
Chartered & Registered Patent Agents,
12, Church Street, Liverpool, and
50, Lincoln's Inn Fields, London,
W.C. 2,

Agents for the Applicants.

COMPLETE SPECIFICATION.

Improvements in the Production of Phosphorus Oxychloride.

We, JOHN STANLEY DUNN and FRANK BRIERS, both British Subjects, both of Norton Hall, The Green, Norton-on-Tees, County Durham, and IMPERIAL CHEMICAL INDUSTRIES LIMITED, a British Company, of Imperial Chemical House, Millbank, London, S.W. 1, do hereby declare the nature of this invention and in what

manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

It is known that when pure calcium orthophosphate is treated with phosgene at a temperature of 325—500° C., phosphorus oxychloride distils over.

We have found that when most

naturally occurring phosphates of calcium are treated with phosgene no substantial production of phosphorus oxychloride ensues. We have also found, however, 5 that this absence of reaction is due to the formation of calcium chloride formed initially by the action of the phosgene upon the other calcium compounds present in the material, notably the carbonate. 10 Based upon this observation, the invention consists in a method of obtaining from naturally occurring phosphates of calcium such as phosphate rock and the like, phosphorus oxychloride by treating the 15 material to remove such other calcium compounds as would react with the phosgene and thereupon submitting the purified calcium phosphate to the action of phosgene or its equivalents at a raised 20 temperature.

For instance, phosphate rock containing 33 per cent. of phosphoric anhydride and 52 per cent. of lime. may be treated with phosgene at a temperature of about 25 300° C. until the carbonate present has been converted into chloride. During this treatment the termination of which may be ascertained by the disappearance of carbon dioxide from the exit gases, no 30 formation of phosphorus oxychloride is observed. The mass is then washed with water to remove calcium chloride and the washed and dried material is subjected to the renewed action of phosgene. An 35 immediate production of phosphorus oxychloride occurs and up to 40 per cent. of the phosphate may be converted by the continued passage of phosgene. When the production of oxychloride slackens the 40 mass may be leached with water to remove further calcium chloride formed by the reaction and the recovered phosphate used again.

The impurities in the crude phosphatic material may also be removed in other 45 ways, for example by treatment with chlorine itself, following by leaching, or the crude material may be washed with a dilute acid such as hydrochloric acid or 50 nitric acid.

Instead of using phosgene in the above process we may use its known equivalents. For example a mixture of carbon monoxide and chlorine, or even chlorine 55 alone, may be passed over a mixture of phosphatic material with activated carbon which secures the formation of phosgene in situ. The carbon may be 60 activated by preliminary treatment with chlorine at a high temperature, e.g. 1000° C. after which it is washed and dried before mixing with the raw material.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to 65 be performed, we declare that what we claim is:—

1. In the production of phosphorus oxychloride by treatment of calcium phosphate with phosgene or its equivalents subjecting natural calcium phosphate to a preliminary treatment to 70 remove other calcium compounds which would react with the phosgene. 75

2. A process as claimed in claim 1 in which the preliminary treatment is effected by phosgene itself after which the material is washed and dried.

3. Process of manufacturing phosphorus oxychloride substantially as described. 80

Dated this 5th day of June, 1930.

W. P. THOMPSON & Co.,
12, Church Street, Liverpool,
Chartered & Registered Patent Agents,